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Indian Standard

SPECIFICATION FOR CALCIUM CHLORIDE

(Second Revision)

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Indian Standard
SPECIFICATION FOR CALCIUM CHLORIDE
(Second Revision)

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Indian Standard

SPECIFICATION FOR CALCIUM CHLORIDE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 25 January 1984, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Calcium chloride, technical, hydrous and anhydrous were covered in IS : 1334-1958* and IS : 1314-1958, respectively. The two standards were amalgamated and revised in 1967 and consequently IS : 1334-1958* was withdrawn. Analytical reagent grades for both were also added in the first revision.

0.3 Calcium chloride, technical, is used by the refrigeration and ice manufacturing industries for making brine and in the chemical industry as a raw material. It is also used for the purpose of spraying on road surfaces as an accelerative for concrete and in curing of concrete. Magnesium compounds are undesirable as refrigerating brines due to their sludge-forming properties, hence in the first revision more stringent limits for magnesium had been specified. Anhydrous calcium chloride, technical, is used as a dehydrating agent in laboratory and a number of industries.

0.4 The standard has been revised once again in the light of the progress made by the industry. In the second revision, with the addition of the fused calcium chloride, three types of the material have been covered. Three grades have been specified for the hydrous material. The two grades which were prescribed for anhydrous material in the first revision have now been merged into one.

0.5 This standard contains clauses **3.2** and **4.2** which permit the purchaser to use his option for selection to suit his requirements.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for calcium chloride technical.

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for calcium chloride.

2. TYPES AND GRADES

2.1 Types — There shall be three types of the material, namely:

a) *Type A* — Hydrated calcium chloride (*see also* **2.2.1**).

b) *Type B* — Anhydrous calcium chloride suitable as a drying agent in analytical work and in various industries, and

c) *Type C* — Fused calcium chloride suitable for use in cement, paint and as a desiccant.

2.2 Grades

2.2.1 Type A of the material, that is, hydrated calcium chloride shall be of the following grades:

a) *Grade* 1 — Suitable as analytical reagent;

b) *Grade* 2 — Lye suitable for refrigeration and chemical industry; und

c) *Grade* 3 — Suitable for use as refrigerant in ice manufacture and as raw material in chemical industries also suitable for use for road construction purposes, for acceleration and curing of concrete.

3. REQUIREMENTS

3.1 Description — The material shall be in the form or flakes or lumps, white in colour and free from extraneous impurities. All forms are deliquescent.

3.1.1 Type A, Grade 1 material may also be in the form of pellets, granules or sticks.

3.2 Size of Lumps — The size of lumps for Type B and Type C material shall be as agreed to between the purchaser and the supplier.

3.3 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. References to the relevant clauses of Appendix A are given in col 8 of Table 1.

3.4 Rejection — The material shall be rejected if it becomes caked or sticks to the containers before opening except for Type A, Grade 3.

TABLE 1 REQUIREMENTS FOR CALCIUM CHLORIDE

(*Clause* 3.3)

Sl No.	Characteristic	Requirements					Method of Test (Ref to Cl No. in Appendix A)
		Type A			Type B	Type C	
		Grade 1	Grade 2	Grade 3			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Matter insoluble in water, percent by mass, *Max*	0·01	0·3	0·4	0·05	0·4	A-2
ii)	Calcium chloride (as $CaCl_2$), percent by mass, *Min*	74·0	35·0	70·0	96·0	75·0	A-3
iii)	Magnesium chloride (as $MgCl_2$), percent by mass, *Max*	—	0·5	0·7	0·5	0·7	A-4
iv)	Alkalinity [(as $Ca(OH)_2$], percent by mass, *Max*	—	0·2	0·3	0·02	0·3	A-5
v)	Chlorides (as NaCl), percent by mass, *Max*	—	0·6	1·6	1·6	1·6	A-6
vi)	Magnesium and alkali salts (as sulphates), percent by mass, *Max*	—	—	—	0·5	—	A-7
vii)	Oxidizing substances (as NO_3), percent by mass, *Max*	0·003	—	—	—	—	A-8
viii)	Sulphates (as SO_4), percent by mass, *Max*	0·010	—	—	0·020	—	A-9
ix)	Ammonium salts (as NH_4), percent by mass, *Max*	0·005	—	—	—	—	A-10
x)	Barium compounds (as Ba), percent by mass, *Max*	0·005	—	—	—	—	A-11
xi)	Heavy metals (as Pb), percent by mass, *Max*	0·005	—	—	0·005	—	A-12

(*Continued*)

TABLE 1 REQUIREMENTS FOR CALCIUM CHLORIDE — *Contd*

Sl No.	Characteristic	Requirements					Method of Test (Ref to Cl No. in Appendix A)
		Type A			Type B	Type C	
		Grade 1	Grade 2	Grade 3			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
xii)	Iron (as Fe), percent by mass, *Max*	0·001	—	—	0·001	—	A-13
xiii)	Magnesium (as Mg), percent by mass, *Max*	0·005	—	—	—	—	A-14
xiv)	Potassium compounds (as K), percent by mass, *Max*	0·01	—	—	—	—	A-15
xv)	Sodium salts (as Na) percent by mass, *Max*	0·02	—	—	—	—	A-16
xvi)	Strontium salts (as Sr), percent by mass, *Max*	0·1	—	—	—	—	A-17
xvii)	*p*H of a 5 percent solution at 25°C	4·5-8·5	7-8·5	7-8·5	7-8·5	7-8·5	A-18

4. PACKING

4.1 Type B and Type C of the material shall be packed in flakes or lumps in moisture-proof bags or steel drums.

4.2 Type A, Grade 1 of the material shall be packed in glass container provided with suitable lids to keep away moisture.

4.3 Type A, Grade 2 of the material shall be packed in glass/polyethylene, carbuoys or shall be transported in tanker.

4.4 Type A, Grade 3 shall be packed in moisture-proof container as agreed to between the purchaser and the supplier.

5. MARKING

5.1 The containers shall be securely closed and legibly and indelibly marked with the following information:

a) Name, type and grade of the material;
b) Name of the manufacturer, and his trade-mark, if any;
c) Net mass of the material; and
d) Lot number in code or otherwise.

5.1.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

6.1 The methods for drawing representative samples of the material and for determination of their conformity with the requirements of this standard shall be as given in Appendix B.

APPENDIX A

(*Clause* 3.3; and *Table* 1)

METHODS OF TEST FOR CALCIUM CHLORIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-2.0 Outline of the Method — A known amount of the sample is dissolved in water and any insoluble matter present is filtered. The residue is washed and dried to constant mass.

A-2.1 Procedure — Transfer quickly about 20 g of the material from the bottle to a tared glass-stoppered weighing bottle, stopper and weigh. Transfer the weighed material to a breaker and dissolve in about 250 ml of water. Filter the solution through a tared Gooch or sintered glass crucible (G No. 4) and wash the residue free from soluble salts. Collect the filtrate and washings in a 500-ml graduated flask and make up the volume with water. Use this solution for tests prescribed in **A-6** and **A-15.2**.

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*Specification for water for general laboratory use (*second revision*).

Dry the crucible along with the residue to constant mass at a temperature of 105 to 110°C.

A-2.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100\,M_1}{M_2}$$

where

M_1 = mass in g of the residue, and
M_2 = mass in g of the material taken for the test.

A-3. DETERMINATION OF CALCIUM CHLORIDE

A-3.0 Outline of the Method — The solution containing a known amount of the sample is titrated against EDTA solution using calcein as indicator.

A-3.1 Reagents

A-3.1.1 *Standard Disodium Ethylenediaminetetraacetate (EDTA) Solution* — Weigh 3·72 g of disodium ethylenediaminetetraacetate dihydrate, dissolve in water and dilute in a graduated flask to 1 litre. Standardize the solution against the standard calcium chloride solution according to the procedure given in **A-3.2.**

A-3.1.2 *Standard Calcium Chloride Solution* — Dissolve 1·0 g of calcium carbonate, dried at 120°C to constant mass in the minimum quantity of concentrated hydrochloric acid (*see* IS : 265-1976*) and then make up the volume to 1 litre. One millilitre of this solution is equivalent to 0·000 4 g of calcium.

A-3.1.3 *Eriochrome Black T Indicator* — Dissolve 0·1 g of the material in 20 ml of rectified spirit. This solution shall be prepared fresh every week.

A-3.1.4 *Calcein Indicator* — Mix 0·01 g of calcein thoroughly with 1 g of potassium chloride.

A-3.1.5 *Buffer Solution* — Dissolve 67·5 g of ammonium chloride in a mixture of 520 ml of ammonium hydroxide (sp gr 0·90) and 250 ml of water. Dissolve a mixture of 0·931 g of EDTA and 0·616 g of magnesium sulphate heptahydrate ($MgSO_4 7H_2O$) in 50 ml of water, combine both the solutions and make up to 1 litre. Five millilitres of buffer solution added to 50 ml of water should not consume more than one drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

A-3.1.6 *Standard Sodium Hydroxide Solution* — 2·5 N.

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*Specification for hydrochloric acid (*second revision*).

A-3.2 Standardization of EDTA Solution — Standardize as given below:

a) *With Calcein Indicator* — Pipette out 50 ml of standard calcium chloride solution (*see* Note) in a 250-ml conical flask and add to it one millilitre of standard sodium hydroxide solution to adjust the *p*H of the solution to about 12. Add 10 mg of calcein indicator and titrate the solution against EDTA solution until the colour changes from yellow-green fluorescence to brown.

b) *With Eriochrome Black T Indicator* — Pipette out 50 ml of standard calcium chloride solution (*see* Note) in 250-ml conical flask and add 5 ml of buffer solution to adjust the *p*H of the solution to approximately 10. Add 5 drops of eriochrome black T indicator and titrate against standard EDTA solution until the wine-red colour changes to pure blue. (The volume of the solution to be titrated against standard EDTA solution should be approximately 50 ml and the strength of the standard EDTA solution should be adjusted in such a way that a similar volume of it is required for the complete titration).

NOTE — The aliquot for titration shall be neutral so that by adding standard sodium hydroxide solution or buffer solution the desired *p*H could be adjusted.

A-3.3 Prepared Sample Solution — Dissolve about 10 g of the material, accurately weighed in water and make up the volume to 1 000 ml.

A-3.4 Procedure — Pipette out 10 ml of the prepared sample solution (*see* **A-3.3**) in a 250-ml conical flask and dilute with 40 ml of water. Add 1·5 ml of standard sodium hydroxide solution followed by 10 mg of calcein indicator. Titrate against the standard EDTA solution till the colour changes from yellow-green fluorescence to brown.

A-3.5 Calculation

$$\text{Calcium (as Ca), percent by mass} = \frac{10\,000\, V_1 A_1}{M}$$

where

V_1 = volume in ml of standard EDTA solution used,

A_1 = mass in g of calcium equivalent to one millilitre of standard EDTA solution as obtained in **A-3.2** (a), and

M = mass in g of the material taken in **A-3.3**.

A-3.5.1 *Calcium Chloride (as $CaCl_2$)* — Deduct the calcium corresponding to calcium hydroxide (*see* **A-5.3**) from the total calcium present and express the balance of calcium as percent calcium chloride (as $CaCl_2$) by multiplying with 2·766.

A-4. DETERMINATION OF MAGNESIUM CHLORIDE

A-4.0 Outline of the Method — The solution containing a known amount of the sample is titrated against EDTA solution using eriochrome black T as indicator.

A-4.1 Reagents

A-4.1.1 *Buffer Solution* — same as given in **A-3.1.5**.

A-4.1.2 *Eriochrome Black T Indicator* — same as given in **A-3.1.3**.

A-4.1.3 *Standard Disodium Ethylenediaminetetraacetate (EDTA) Solution* — same as given in **A-3.1.1**.

A-4.2 Procedure — Pipette out 10 ml of the *prepared sample solution* (*see* **A-3.3**) in a 250-ml conical flask and dilute with 35 ml of water. Add 5 ml of buffer solution and 5 drops of eriochrome black T indicator. Titrate against standard EDTA solution till the wine-red colour of the solution changes to blue. Note the reading of the burette.

A-4.3 Calculation

$$\text{Magnesium, percent by mass} = \frac{6\,068\ (\ V_2A_2 - V_1A_1\ )}{M}$$

where

V_2 = volume in ml of standard EDTA solution used in **A-4.2**,

A_2 = mass in g of calcium equivalent to 1 ml of standard EDTA solution as obtained in **A-3.2**(a),

V_1 = volume in ml of standard EDTA solution used in **A-3.4**,

A_1 = mass in g of calcium equivalent to 1 ml of standard EDTA solution as obtained in **A-3.2**(b), and

M = mass in g of the material taken in **A-3.3**.

A-4.4 Magnesium Chloride (as $MgCl_2$) — Express magnesium present as magnesium chloride (as $MgCl_2$) by multiplying with 3·916.

A-5. DETERMINATION OF ALKALINITY

A-5.0 Outline of the Method — A known amount of the sample is dissolved in freshly boiled and cooled water, filtered and titrated against hydrochloric using phenolphthalein as indicator.

A-5.1 Reagents

A-5.1.1 *Standard Hydrochloric Acid* — 0·1 N.

A-5.1.2 *Phenolphthalein Indicator* — Dissolve 0·1 g of phenolphthalein in 100 ml of rectified spirit.

A-5.2 Procedure — Weigh accurately about 10 g of the sample, dissolve it in 50 ml of freshly boiled and cooled water, filter the solution through folded filter paper, wash the filter paper thoroughly but as quickly as possible and collect the filtrate and washings in a 200-ml conical flask. Titrate the collected solution against standard hydrochloric acid using phenolphthalein as indicator.

A-5.3 Calculation

$$\text{Alkalinity [as } Ca(OH)_2 \text{] percent by mass} = \frac{3{\cdot}705\, VN}{M}$$

where

V = volume in ml of the standard hydrochloric acid used for the titration,

N = normality of standard hydrochloric acid, and

M= mass in g of the material taken for the test.

A-6. DETERMINATION OF ALKALI CHLORIDES

A-6.0 Outline of the Method — An aliquot of the solution of the sample obtained after determination of matter insoluble in water is treated with dilute nitric acid and excess of silver nitrate. The precipitate is filtered and washed. The filtrate together with the washings is titrated with standard thiocyanate solution using ferric ammonium sulphate as indicator.

A-6.1 Reagents

A-6.1.1 *Dilute Nitric Acid* — approximately 5 N.

A-6.1.2 *Standard Silver Nitrate Solution* — 0·1 N.

A-6.1.3 *Standard Ammonium or Potassium Thiocyanate Solution* — 0·1 N.

A-6.1.4 *Ferric Ammonium Sulphate Solution* — approximately 40 percent (*m*/*v*) solution in water.

A-6.2 Procedure — Transfer 50 ml of the solution reserved in **A-2.1** to a 250-ml graduated flask and make up the volume of solution with water. Pipette 50 ml of this solution to a breaker and dilute to 100 ml. Add 5 ml of dilute nitric acid and then add in excess a measured volume of silver nitrate solution. Stir to coagulate the precipitate. Filter, and wash the precipitate with dilute nitric acid. Add to the combined filtrate and washings about 2 to 3 ml of ferric ammonium sulphate solution and titrate the excess of silver nitrate with standard ammonium or potassium thiocyanate solution.

A-6.2.1 Carry out a blank determination at the same time.

A-6.3 Calculation

$$\text{Chloride (as Cl), percent by mass} = \frac{3{\cdot}546\ (V_1 - V_2)\ N}{M}$$

where

V_1 = volume in ml of standard ammonium or potassium thiocyanate solution required for the blank,

V_2 = volume in ml of standard ammonium or potassium thiocyanate solution required for the test with the material,

N = normality of standard ammonium or potassium thiocyanate solution, and

M = mass in g of the material in the aliquot.

A-6.4 Alkali Chlorides (as NaCl) — Deduct the chloride corresponding to calcium chloride and magnesium chloride from the total chloride present and calculate the residue chloride (as NaCl) by multiplying with 1·648 7.

A-7. TEST FOR MAGNESIUM AND ALKALI SALTS

A-7.0 Outline of the Method — The sample is dissolved in water, acidified and heated to boiling. A slight excess of hot ammonium oxalate is added and rendered alkaline with ammonium hydroxide. Cooled and filtered. Filtrate is acidified with sulphuric acid and nitric acid and evaporated to dryness. The residue is dissolved in water and filtered. The combined filtrate and washings are evaporated to dryness and finally ignited to constant mass.

A-7.1 Reagents

A-7.1.1 *Hydrochloric Acid* — 20 percent (*v/v*).

A-7.1.2 *Methyl Red Indicator Solution* — Dissolve 0·15 g of methyl red in 500 ml of water.

A-7.1.3 *Ammonium Oxalate Solution* — Dissolve 35 g of ammonium oxalate [$(NH_4)_2C_2O_4H_2O$] in water and make up to 1 000 ml.

A-7.1.4 *Ammonium Hydroxide Solution* — 10 percent (*v/v*).

A-7.1.5 *Sulphuric Acid* — 10 percent (*v/v*).

A-7.1.6 *Concentrated Nitric Acid* — (*see* IS : 264-1976*).

A-7.2 Procedure — Dissolve 1·0 g of the material in 100 ml of water, add 5 ml of hydrochloric acid and few drops of methyl red indicator solution, and heat to boiling. Add dropwise with stirring 100 ml of warm

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*Specification for nitric acid (*second revision*).

ammonium oxalate solution. Heat the solution to 70-80°C, and add dropwise ammonium hydroxide solution, until it is just alkaline to methyl red. Dilute to 250 ml and allow to cool for 1 hour. Filter, add to 125 ml of the filtrate 5 ml of sulphuric acid, evaporate to about 30 ml, and cool. Add 25 ml of nitric acid and evaporate to dryness on a steam-bath. Dissolve the residue in a small quantity of water, filter through a small filter paper, and wash. Transfer the filtrate and washings to a small tared porcelain basin. Evaporate to dryness and ignite gently to volatilize the excess acids and salts and finally ignite at 800 ± 25°C for 25 minutes. Weigh the tared dish.

A-7.3 The limit specified in Table 1 shall be deemed as not having been exceeded if the ignited residue does not weigh more than 0·010 0 g.

A-8. TEST FOR OXIDIZING SUBSTANCES

A-8.0 Outline of the Method — The material is dissolved in ice cold sulphuric acid. Diphenylamine is added and digested on a steam-bath. The colour produced is compared with the colour produced in a control test.

A-8.1 Reagents

A-8.1.1 *Concentrated Sulphuric Acid* — (*see* IS : 266-1977*).

A-8.1.2 *Diphenylamine Solution* — Dissolve 10 mg of colourless diphenylamine in 100 ml of sulphuric acid. In another beaker dissolve 2 g of ammonium chloride in 200 ml of water. Cool both solutions in ice-bath. Cautiously add the sulphuric acid solution to the water solution, taking care to keep the resulting solution cold.

A-8.1.3 *Standard Nitrate Solution* — Dissolve 0·164 g of potassium nitrate (KNO_3) in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the solution contains 0·01 mg of nitrate (as NO_3).

A-8.1.4 *Sodium Carbonate*

A-8.2 Procedure — Transfer 0·200 g of the sample into a dry beaker. Cool it thoroughly in an ice-bath and add 22 ml of sulphuric acid, already cooled to ice-bath temperature. Allow the mixture to warm to room temperature and swirl the beaker at intervals to effect quiet solution with gentle evolution of hydrogen chloride. When solution is complete, add 3 ml of diphenylamine solution and digest on steam-bath for 90 minutes. In another dry beaker evaporate to dryness a solution containing 0·6 ml of the standard nitrate solution and 0·01 g of sodium carbonate. Treat the residue in the same way as the material.

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*Specification for sulphuric acid (*second revision*).

A-8.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced with the material is not darker than that produced with the standard nitrate solution.

A-9. DETERMINATION OF SULPHATES

A-9.0 Outline of the Method — The solution of the sample which is weakly acid medium is heated to boil for 5 minutes and treated with hot barium chloride solution. The barium sulphate precipitate filtered, washed and ignited to constant mass.

A-9.1 Reagents

A-9.1.1 *Methyl Red Indicator Solution* — same as in **A-7.1.2**.

A-9.1.2 *Ammonium Hydroxide Solution* — free from carbonate.

A-9.1.3 *Concentrated Hydrochloric Acid* — (*see* IS : 265-1976*).

A-9.1.4 *Barium Chloride Solution* — Dissolve 120 g of barium chloride ($BaCl_2 2H_2O$) in water and dilute to 1 litre.

A-9.2 Procedure — Dissolve 10·0 g of the material in 100 ml of water and heat to boiling. Add a few drops of methyl red indicator solution, make alkaline with ammonium hydroxide solution and boil for 5 minutes. Filter through a small filter paper, and wash with a little hot water. Reject the residue. Neutralize the filtrate with hydrochloric acid and add 1 ml in excess. Determine the sulphate content as prescribed in IS : 2317-1975†).

A-9.3 Calculation

$$\text{Sulphates (as } SO_4 \text{), percent by mass} = 41{\cdot}15 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the precipitate, and

M_2 = mass in g of the material contained in the solution taken for precipitation.

A-10. TEST FOR AMMONIUM SALTS

A-10.0 Outline of the Method — The solution of the sample is treated with sodium hydroxide and filtered. To the filtrate, Nessler solution is added and the colour produced is compared with that produced in a control test containing definite amount of ammonium hydroxide.

A-10.1 Reagents

A-10.1.1 *Sodium Hydroxide Solution* — 10 percent (*m/v*).

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*Specification for hydrochloric acid (*second revision*).

†Method for the gravimetric determination of sulphates (*first revision*).

A-10.1.2 *Nessler Solution* — Dissolve 143 g of sodium hydroxide in 700 ml of water. Dissolve 50 g of red mercuric iodide and 40 g of potassium iodide in 200 ml of water. Pour the iodide solution into hydroxide solution and dilute to 1 000 ml. Allow to settle and use the clear supernatant liquid.

A-10.1.3 *Standard Ammonium Chloride Solution* — Dissolve 0·296 g of ammonium chloride (NH_4Cl) in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0·01 mg of ammonium (as NH_4).

A-10.2 Preparation of Test Solution — Dissolve 50·0 g of the material in about 200 ml of water, filter carefully, if necessary, and dilute to 250 ml in a measuring flask (solution A).

A-10.3 Procedure — Dilute 10 ml of the solution A (**A-10.2**) to 45 ml and add 15 ml of sodium hydroxide solution. Filter through a sintered glass crucible, previously washed with sodium hydroxide solution. Take 6 ml of the filtrate, dilute with water to 50 ml and add 2 ml of Nessler solution. In a control test, having an equal volume of the solution and containing 1 ml of standard ammonium chloride solution and 1·5 ml of sodium hydroxide, add 2 ml of Nessler solution.

A-10.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced with the material is not darker than that provided with the standard solution.

A-11. TEST FOR BARIUM SALTS

A-11.0 Outline of the Method — The solution of the sample is treated with sodium acetate, acetic and potassium dichoromate. The turbidity due to barium chromate produced is compared with that produced in a control test.

A-11.1 Reagents

A-11.1.1 *Sodium Acetate* — solid.

A-11.1.2 *Acetic Acid*

A-11.1.3 *Standard Barium Solution* — Dissolve 0·178 g of barium chloride ($BaCl_2 2H_2O$) in water and dilute to 1 000 ml.

A-11.1.4 *Potassium Dichromate Solution* — Dissolve 10 g of potassium dichromate ($K_2Cr_2O_7$) in water and dilute to 100 ml with water.

A-11.2 Procedure — Take in a tall beaker 15 ml of solution A (**A-10.2**) and add to it 2 g of sodium acetate and 0·05 ml of acetic acid. Add 2 ml of potassium dichromate solution and allow to stand for 15 minutes.

In another tall beaker take 5 ml of standard barium solution, and add to it 2 g of sodium acetate, 0·05 ml of acetic acid and dilute to 15 ml. Add 2 ml of potassium dichromate solution, and allow to stand for 15 minutes.

A-11.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than that produced in the control test.

A-12. TEST FOR HEAVY METALS

A-12.0 Outline of the Method — The solution of the sample in weakly acetic acid medium is treated with hydrogen sulphide water and the colour produced is compared with that produced with a standard lead solution.

A-12.1 Reagents

A-12.1.1 *Acetic Acid* — 1 N.

A-12.1.2 *Standard Lead Solution* — Dissolve 0·160 g of lead nitrate [$Pb(NO_3)_2$] in 100 ml of dilute nitric acid (1 : 99) and dilute to 1 000 ml. Take 20 ml of this solution and dilute to 100 ml with water. One millilitre of this diluted solution contains 0·2 mg of lead (as Pb).

A-12.1.3 *Hydrogen Sulphide Water* — Saturate water with hydrogen sulphide. This solution shall be freshly prepared.

A-12.2 Procedure — Take 30 ml of solution A (**A-10.2**) in a Nessler cylinder (B), add 1 ml of acetic acid and dilute to 40 ml. In another Nessler cylinder (C) take 1 ml of standard lead solution, add to it 10 ml of solution A (**A-10.2**) and 1 ml of acetic acid, dilute to 40 ml. Add 10 ml of hydrogen sulphide water to each. Dilute the contents of each cylinder to 50 ml and mix well.

A-12.3 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in cylinder B is not more intense than that in cylinder C.

A-13. TEST FOR IRON

A-13.0 Outline of the Method — The solution of the sample is oxidized with ammonium persulphate and treated with ammonium thiocyanate. The colour produced is compared with that produced with a standard iron solution.

A-13.1 Reagents

A-13.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-13.1.2 *Dilute Sulphuric Acid* — approximately 5 N.

A-13.1.3 *Ammonium Persulphate* — solid.

A-13.1.4 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient N-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-13.1.5 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] in 10 ml of 10 percent sulphuric acid and dilute to 100 ml with water. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the solution contains 0·01 mg of iron (as Fe).

A-13.2 Procedure — Take 10 ml of the solution A (**A-10.2**) and dilute with 20 ml of water and transfer to a Nessler cylinder. Add 5 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Dilute to the mark with water. Shake vigorously for 30 seconds and allow the liquids to separate. Carry out a control test in the other Nessler cylinder with 1 ml of standard solution.

A-13.3 The limits prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-14. TEST FOR MAGNESIUM

A-14.0 Outline of the Method — The solution of the sample is treated with titan yellow solution and sodium hydroxide. The colour developed is compared with that produced with a standard magnesium solution.

A-14.1 Reagents

A-14.1.1 *Sodium Hydroxide Solution* — approximately 1 N.

A-14.1.2 *Titan Yellow Solution* — 0·1 percent solution in water.

A-14.1.3 *Standard Magnesium Solution* — Dissolve 1·014 g of clear crystals of magnesium sulphate heptahydrate ($MgSO_4 \cdot 7H_2O$) in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this diluted solution is equivalent to 0·01 mg of magnesium (as Mg).

A-14.2 Procedure — Take in a Nessler cylinder (B) 7·5 ml of solution A (**A-10.2**) and dilute to 20 ml. Add to it 0·15 ml of the titan yellow solution and then 2 ml of sodium hydroxide solution. Mix well and allow to stand for 10 minutes after which dilute to 50 ml and mix well. In another Nessler cylinder (C) take 2·5 ml of solution A (**A-10.2**) and add to it 5 ml of standard magnesium solution and dilute to 20 ml. Treat it also in the same way as described above. Compare the colour developed in both the cylinders.

A-14.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour in cylinder B is not darker than that in cylinder C.

A-15. DETERMINATION OF POTASSIUM

A-15.0 Outline of the Method — Potassium is determined instrumentally by the use of a flame photometer. Alternatively the solution of the sample at ice cold temperature is treated with sodium tetraphenyl boron. The precipitate is filtered, washed and dried at 110°C.

A-15.1 Flame Photometer Method

A-15.1.1 *Apparatus*

A-15.1.1.1 *Flame photometer* — with red-sensitive photo-tube.

A-15.1.1.2 *Oxy-hydrogen burner*

A-15.1.2 *Reagents*

A-15.1.2.1 *Concentrated hydrochloric acid* — (*see* IS : 265-1976*).

A-15.1.2.2 *Standard potassium chloride solution* — Dissolve 0·191 g of potassium chloride (KCl) in water and dilute to 1 000 ml. Take 100 ml of this solution and dilute to 1 000 ml with water. One millilitre of this diluted solution is equivalent to 0·01 mg of potassium (as K).

A-15.1.3 *Procedure*

A-15.1.3.1 *Preparation of solutions*

Sample solution S — To a 5-ml aliquot of solution *A* (**A-10.2**), add 1 ml of hydrochloric acid and dilute to 100 ml with water.

Control solution C — To another 5-ml aliquot of solution *A* (**A-10.2**), add 1 ml of standard potassium chloride solution, 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-15.1.3.2 Set the flame photometer according to the directions of the manufacturer of the apparatus. Observe the maximum intensity of radiation produced by control solution C at 767 nm (potassium line). Observe the intensity of radiation produced by sample solution *S* at 767 nm and also at 750 nm with the same adjustments of the instrument.

A-15.1.3.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the difference (D_1) between the intensities observed at 767 nm and 750 nm for sample solution *S* does not exceed the differnce (D_2) between the intensities observed at 767 nm for control solution *C* and sample solution *S*.

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*Specification for hydrochloric acid (*second revision*).

A-15.2 Sodium Tetraphenylboron Method

A-15.2.1 *Apparatus*

A-15.2.1.1 *Sintered glass crucible* — G No. 4 or Gooch crucible.

A-15.2.1.2 *Ice-bath*

A-15.2.2 *Reagents*

A-15.2.2.1 *Sodium tetraphenylboron* — Dissolve 1 g of sodium tetraphenylboron in 100 ml of 0·01 N sodium hydroxide solution. Before use cool the solution in ice.

A-15.2.2.2 *Wash solution* — Recrystallize potassium tetraphenylboron from acetone and saturate water with the recrystallized product. The solution shall be prepared fresh every day.

A-15.2.3 *Procedure* — Transfer 25 ml of the solution reserved in **A-2.1** into a 100-ml conical flask. Place the flask in an ice-bath for 5 to 10 minutes. When the solution has cooled, add 25 ml of sodium tetraphenylboron solution previously cooled in ice. Mix by gentle swirling and leave in the ice-bath for 5 to 10 minutes. Filter through a sintered glass crucible (G No. 4) or a Gooch crucible, wash the flask and the precipitate with three 10 ml portions of the wash solution and dry the crucible containing the precipitate for one hour at 110-120°C. Cool in a desiccator and weigh.

A-15.2.4 *Calculation*

Potassium (as K), percent by mass (on dry basis)

$$= \frac{21\,820\,M_1}{M_2 9\,(\,100 - M\,)}$$

where,

M_1 = mass in g of the precipitate,

M_2 = mass in g of the prepared sample taken for the test in **A-2.1**, and

M = percent moisture content.

A-16. TEST FOR SODIUM

A-16.0 Outline of the Method — Sodium is determined instrumentally by the use of a flame photometer.

A-16.1 Apparatus

A-16.1.1 *Flame Photometer* — same as in **A-15.1.1.1**.

A-16.1.2 *Oxy-Hydrogen Burner* — same as in **A-15.1.1.2**.

A-16.2 *Reagents*

A-16.2.1 *Cocentrated Hydrochloric Acid* — (*see* IS : 265-1976*).

A-16.2.2 *Standard Sodium Chloride Solution* — Dissolve 0·254 g of sodium chloride (NaCl), dried at 105 ± 5°C in water and dilute to 1 000 ml. One millilitre of the solution contains 0·1 mg of sodium (as Na).

A-16.3 Procedure

A-16.3.1 *Preparation of Solution*

A-16.3.1.1 *Sample solution S* — To a 5-ml aliquot of Solution *A* (**A-10.2**), add 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-16.3.1.2 *Control solution C* — To another 5-ml aliquot of solution *A* (**A-10.2**), add 2 ml of standard sodium chloride solution and 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-16.4 Procedure — Set the flame photometer according to the directions of the manufacturer of the apparatus. Observe the emission of control solution *C* at 589 nm (sodium line) and the emission of sample solution *S* at 589 nm and 580 nm.

A-16.5 The limit prescribed in Table 1 shall be taken as not having been exceeded if the difference (D_1) between the intensity observed for sample solution *C* at 580 nm does not exceed the difference (D_2) observed at 589 nm between sample solution *S* and control solution *C*.

A-17. TEST FOR STRONTIUM

A-17.0 Outline of the Method — Strontium is determined instrumentally by the use of a flame photometer.

A-17.1 Apparatus

A-17.1.1 *Flame Photometer* — same as in **A-15.1.1.1**.

A-17.1.2 *Oxy-Hydrogen Burner* — same as in **A-15.1.1.2**.

A-17.2 Reagents

A-17.2.1 *Concentrated Hydrochloric Acid* — (*see* IS : 265-1976*).

A-17.2.2 *Standard Strontium Nitrate Solution* — Dissolve 0·242 g of strontium nitrate [$Sr(NO_3)_2$] in water and dilute to 100 ml. One millilitre of this solution contains 0·01 mg of strontium (as Sr).

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*Specification for hydrochloric acid (*second revision*).

A-17.3 Procedure

A-17.3.1 *Preparation of Solutions*

A-17.3.1.1 *Sample solution S* — To a 5-ml aliquot of solution *A* (**A-10.2**), add 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-17.3.1.2 *Control solution C* — To another 5-ml aliquot of solution *A* (**A-10.2**), add 10 ml of standard strontium nitrate solution and 1 ml of hydrochloric acid, and dilute to 100 ml with water.

A-17.3.2 Set the flame photometer according to the directions of the manufacturer of the apparatus. Observe the emission of control solution *C* at 460·7 nm (strontium line) and the emission of sample solution *S* at 460·7 nm and a wave length of 2 or 4 nm less than 460·7 nm.

A-17.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the difference (D_1) between the intensity observed for sample solution *S* at 460·7 nm and 460·7 nm −2 nm or −4 nm does not exceed the difference (D_2) observed at 460·7 nm between the solution *S* and solution *C*.

A-18. DETERMINATION OF *p*H

A-18.0 Outline of the Method — *p*H is determined instrumentally by the use of a *p*H meter.

A-18.1 Apparatus

A-18.1.1 *p*H *Meter* — Any suitable *p*H meter equipped with a standard calomel electrode and glass electrode. The meter shall be accurate to 0·05 *p*H unit in the *p*H range of 4·01 to 9·18.

A-18.2 Procedure — Dissolve 10 g of the material in 200 ml of water at 25°C. Determine the *p*H in accordance with the directions provided by the manufacturer.

APPENDIX B

(*Clause* 6.1)

SAMPLING OF CALCIUM CHLORIDE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, storing, preparing and handling test samples the following precautions shall be observed.

B-1.1 Samples shall not be taken at a place which is exposed to weather.

B-1.2 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in suitable dry and air-tight containers.

B-1.5 Each sample container shall be sealed air-tight after filling and shall be marked with full details of sampling, the date of sampling and the year of manufacture.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of calcium chloride of the same type and grade drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

If the material is stored in large drums, each drum will constitute a lot.

B-2.2 The number of containers (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE (N)	NUMBER OF CONTAINERS TO BE SELECTED (n)
4 to 50	3
51 ,, 100	4
101 ,, 150	5
151 ,, 300	7
301 and above	10

NOTE — When the size of the lot is 3 or less, the number of containers to be selected and the criterion for judging conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them in-one order as 1, 2, 3, ... up to r and so on, where r is the integral part of N/n (N and n being the lot size and sample size, respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

B-2.4 Samples shall be tested for each lot for ascertaining the conformity of the material to the repuirements of this specification.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the *n* containers selected according to **B-2.3**, a portion of the material, about 200 g, shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions, equal quantities of the material shall be drawn and mixed thoroughly to form a composite sample of about 500 g. The composite sample shall be divided into three parts, one for the purchaser, another for the supplier and the third used as a referee sample.

B-3.2.1 If the lot consists of a single drum, composite sample shall be formed by mixing the material drawn from a number of places in the drum with suitable sampling instrument.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.4 All the individual samples and the composite sample shall be transferred to separate sampling containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier and shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 For all the different types and grades of the material, tests for the determination of calcium chloride content shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all other characteristics given in Table 1 shall be performed on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results, the average (X) for the calcium chloride content and the range (R) shall be computed (range R being defined as the difference between the maximum and the minimum values of the test results). The appropriate expression as shown in col 6 of Table 3 shall be calculated for the characteristic. If the values of the expression satisfy the conditions in col 6 of Table 3, the lot shall be declared to have satisfied the requirement for calcium chloride content.

TABLE 3 CRITERIA FOR CONFORMITY FOR CALCIUM CHLORIDE CONTENT

SL No.	TYPE	GRADE	AVERAGE OF TEST RESULTS 1, 2, 3, ... n	RANGE	CRITERIA FOR CONFORMITY
(1)	(2)	(3)	(4)	(5)	(6)
i)	A	1	$\bar{X}_1$	R_1	($\bar{X}_1$ – 0·6 R_1) shall be greater than or equal to 74·0
ii)	A	2	$\bar{X}_2$	R_2	($\bar{X}_2$ – 0·6 R_2) shall be greater than or equal to 70·0
iii)	A	3	$\bar{X}_3$	R_3	($\bar{X}_3$ – 0·6 R_3) shall be greater than or equal to 70·0
iv)	B	—	$\bar{X}_4$	R_4	($\bar{X}_4$ – 0·6 R_4) shall be greater than or equal to 96·0
v)	C	—	$\bar{X}_5$	R_5	($\bar{X}_5$ – 0·6 R_5) shall be greater than or equal to 90·0

B-5.2 For Composite Samples — The lot shall be deemed to have met the requirements of the characteristics tested on the composite sample if the test results satisfy the corresponding requirements given in Table 1.

B-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **B-5.1** and **B-5.2**.

(*Continued from page* 2)

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